ASBESTOS IN SOIL/ROCK

El Dorado County Site Discovery

A. Why are the percent asbestos results reported by Lab 2 following CARB 435 consistently elevated over the percent asbestos results by Lab 2 following EPA Method 600/R-93/116 (TEM)?

The CARB 435 method reports results in numerical percent asbestos while the EPA method reports weight percent asbestos. As outlined below, these methods can yield quite varied results, even when the procedures are followed precisely as written.

The CARB 435 method uses point counting to quantify asbestos in soil samples. In this procedure, eight slides are prepared and analyzed with a polarized light microscope (PLM). The eyepiece of the PLM contains either a crosshair or other type of graticule (e.g., Chalkley point array) that superimposes itself over the image of the sample during the analysis. The analyst counts 50 particles (non-empty points) per slide that fall under the crosshair intersection or array point(s) and categorizes them as asbestos or non-asbestos, based on morphology and optical properties. After 400 points have been counted, the concentration of asbestos is calculated as number of asbestos points / total points and is reported as percent asbestos.

Lab 2 uses a crosshair to point-count CARB 435 samples. Starting from one edge of the preparation, the analyst identifies each particle that falls under the crosshair as asbestos or non-asbestos. The analysis proceeds in a random manner across each slide until the requisite number of particles is counted.

The method specifically states that if a particle falls under more than one point using a Chalkley array or cross-hair graticule, it is only to be counted once. Therefore, there is no way for the method to account for the differences in size between asbestos and non-asbestos particles. When counted, small asbestos fiber bundles and large non-asbestos soil particles contribute equally to the CARB 435 total particle count, even though the area occupied by each particle may be obviously unequal.

This problem is compounded further by the fact that the PLM image is two-dimensional and the analysis does not consider the depth, volume or density of the particles counted. To illustrate the severity of this problem, the results of a hypothetical point count are interpreted below by several analytical methods. Consider a simplified PLM point-count analysis in which 40 chrysotile asbestos fibers and 360 spherical quartz particles (a common soil mineral) are counted. If all these particles were counted in a CARB 435 analysis, the asbestos concentration would be calculated as:

40 asbestos points / 400 non-empty points = 10 *numerical* percent asbestos.

But if the size of these particles is considered, the actual asbestos content can be substantially less. For ease of calculation, assume each fiber is ten microns long and one micron in diameter and each sphere is ten microns in diameter. If quantifying by visual estimation, the analyst estimates the area occupied by asbestos fibers relative to the area occupied by total particulate (including asbestos fibers). In this example, each fiber occupies 10 μm^2 and quartz sphere occupies 79 μm^2. Therefore, the concentration of asbestos in this analysis is:

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(10)(40)/[(79)(360)+(10)(40)] = 1.4 *area* percent.

However, we are not counting flat particles. Each particle has a depth that must be considered when calculating volume or weight percent. In this example, the approximate volume of each cylindrical fiber is 8 μm^3 and each sphere is 524 μm^3. Therefore, the concentration of asbestos in this analysis is:

(8)(40)/[(524)(360)+(8)(40)] = 0.17 *volume* percent.

Since chrysotile and quartz have essentially the same densities (2.55 and 2.65 g/cm^3, respectively), volume percent is approximately equal to *weight* percent. In a soil containing high levels of hematite, magnetite or other relatively dense non-asbestos minerals, this problem only gets worse. If the non-asbestos particle density doubles, the calculated weight percent of asbestos in the above example is halved. However, this idealized sample, that was point counted at 10 numerical percent, is actually only 0.17 weight percent asbestos. The CARB method overestimated the asbestos content in this hypothetical sample by nearly sixty times *on the same counted particles*. In light of the above discussion, the results from the PLM and TEM analyses are for the most part within acceptable limits. A brief analysis of non-asbestos particles in some of the samples from this project found them to be mostly magnesium silicates (i.e.,lizardite), aluminum silicates (i.e., clay, feldspar), and quartz; so, the error estimates given in the above example could occur in these samples.

The differences between numerical percent and weight percent in the above example should be considered a minimal estimate of the error seen with point counting soil samples. Uniform ten-micron quartz particles were used for the example. In fact, particles of various sizes up to 74 µm in diameter may be present after 200-mesh sieving. These larger non-asbestos particles would contribute larger volumes to the denominator in the above weight percent calculation, making the weight percent result even lower.

The reported result in a CARB 435 analysis should be given as *numerical percent asbestos* units, since it is derived from particle numbers. Unless all particles, asbestos and non-asbestos, are of the same size and density, there is not really any reliable relationship between numerical percent and *weight percent* units.

The TEM method performed by Lab 2 yields a truer weight percent result by taking into account fiber dimensions, converting them to mass via density, and relating them to the starting mass of sample prepared. There is no comparing of asbestos to non-asbestos components in the sample. A known weight of sample is ashed at 480°C and acid-washed in hydrochloric acid to remove organic and acid-soluble components, respectively. The residue is suspended in a known volume of particle-free water and aliquots of the resulting suspension are filtered over a known filter area. The filters are prepared directly and mounted on TEM grids having predetermined grid opening areas. A known number of grid openings are analyzed at low and high magnification to ensure that both large and small asbestos fibers are included in the analysis, if present. Detected asbestos fibers are identified by morphology, selected area electron diffraction (SAED), and/or energy dispersive x-ray spectroscopy (EDX). As part of the analysis, the length and diameter of each detected asbestos fiber is recorded. Asbestos fibers in complex structures are counted and sized individually.

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In the first step of TEM data reduction, the volume, V, of each detected fiber is calculated using the standard geometric equation $V=\pi R^2 L$, where R is the fiber radius and L is the fiber length. Multiplying the fiber volume by its known density gives its mass. The masses of fibers counted at high and low magnifications are summed and normalized to the aliquot volume and number of grid openings analyzed in the low magnification scan, resulting in a total mass analyzed on the grid. Back-calculating through the grid opening area, effective filter area, gravimetric suspension volume and starting weight gives a result of mass asbestos/starting sample mass, or weight percent asbestos. Again, note that there is no comparison between asbestos and non-asbestos particles, so any size differential is irrelevant.

Another possible source of the noted differences may be sample non-homogeneity. While the samples were visually well mixed after milling, homogeneous particulate dispersion on a microscopic scale is very difficult to assess or confirm. Unlike liquid solutions, which are easily made homogeneous by simple stirring, particles are notoriously difficult to mix evenly in a dry matrix, even with the use of milling techniques. Factor in the wide range in particle size, from 74 µm (200-mesh sieve) particles to 0.05 µm diameter chrysotile fibers, and particle distribution becomes even more of an issue.

B. Why are the percent asbestos results reported by Lab 2 consistently elevated over results from Lab 1 when both followed CARB 435 method on the same 18 samples?

The two laboratories in question have had no direct contact over this issue, so this discussion will, of necessity, include some speculation as to specific details regarding the way Lab 1 performed its CARB 435 analysis on these samples. While the published method gives step by step procedures to complete the analysis, subtle variations are allowed that can result in varied results from different laboratories.

One such variation is the type of graticule used in the microscope's eyepiece to perform point counting. The graticule used by Lab 2 contains a crosshair that delineates a single point. Particles falling under the intersection of its two perpendicular lines are identified and counted as asbestos or non-asbestos. The slide is moved at least fifty times (assuming some empty points) to obtain a fifty-particle-count per slide preparation, so a relatively large portion of the sample is observed. Eight slide preparations are counted to give a 400-particle point count. The Chalkley point array is another permissible type of graticule, and it may have been used by Lab 1. It contains an irregular, fixed arrangement of 25 or 100 points that are superimposed on the image of the sample. Asbestos and non-asbestos points are identified and counted as above, but the analysis of a heavily loaded slide can be completed after just one to two fields of view per slide. If the asbestos fiber distribution on the slide is non-uniform, as is commonly the case with soil samples, the Chalkley array could have a greater tendency to miss asbestos points, or conversely, hit a "hot spot" and give an artificially high count.

The largest absolute differences in the reported asbestos content between the two laboratories appear where Lab 2 results are above ten percent. For these samples, Lab 2 exercised Exception 2 found in section 8.3 of the method: "If the sample is suspected to have an asbestos content in excess of ten percent, a visual technique can be used to report that the sample contains greater than ten percent asbestos." A footnote to this effect was printed on

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Lab 2's report for each of these samples. Of the eighteen samples analyzed by both laboratories, Lab 2 reported five at greater than ten percent using visual estimation to determine asbestos content. The results from the same samples from Lab 1 indicate that they counted them by point counting.

Section 8.2 of the method states that "positive identification of asbestos requires the determination of the following optical properties: morphology, color and pleochroism, refractive indices, birefringence, extinction characteristics, and sign of elongation." Section 8.3 further states that "all optical properties in section 8.2 shall be determined to positively identify asbestos," and "the analyst, in addition to performing the particle count, will also determine if the fiber is asbestos or not." The determination of refractive index may be performed by either dispersion staining or Becke line procedure, both of which require the sample to be mounted in the appropriate refractive index (RI) liquid, which is 1.550 for chrysotile asbestos.

It appears that the two laboratories used different means to identify asbestos fibers in their respective CARB 435 analyses. Lab 2 mounted all samples in 1.550 RI oil and identified and counted particles on these mounts. The refractive indices, in both the parallel and perpendicular directions, of all counted chrysotile fibers were determined using the dispersion staining technique. (This is another advantage of using a crosshair graticule--all particles are centered on the optical axis of the microscope, so rotating the stage to observe dispersion staining colors is simplified. Asbestos fibers under off-axis Chalkley array points must be rotated to confirm their refractive indices, and then moved back to the point under which they were counted, a much more awkward and time-consuming technique.)

However, in a written description of their analytical procedure, Lab 1 stated that they identified chrysotile asbestos fibers in 1.55 RI oil, but performed point counting on samples mounted in a different RI liquid. Technically speaking, they should not have reported *any* chrysotile counts, since they could not confirm the refractive indices of the fibers they counted. In a non-matching RI oil, Lab 1 could only use morphology to differentiate asbestos from non-asbestos particles. All the other optical properties noted above are skewed in an inappropriate RI oil. The statement by Lab 1 that using non-matching oils for this method "is a standard procedure used by all PLM analysts" may apply to all PLM analysts employed by Lab 1, but definitely does not apply to analysts in Lab 2 or other labs that were consulted on this issue. If the fibers counted in a point count analysis are to be identified, they must be mounted in the appropriate RI oil.

The definition of a countable fiber is another issue that can cause varied counts between laboratories. The CARB 435 method states that "For the purposes of the method, 'asbestos fibers' are defined as mineral fibers having aspect ratios [length to width] greater than or equal to 3:1...." Many geologists and mineralogists use a much higher limit in their fiber definition, up to 20:1 to 100:1, and Lab 1 analysts may as well. However, the method permits fibers with aspect ratios as low as 3:1 to be counted, and Lab 2 followed this definition. Counting lower aspect ratio fibers as asbestos would elevate results from Lab 2 over those from Lab 1.

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